Historic, archived document

Do not assume content reflects current scientific knowledge, policies, or practices.

P69C

SEP 4 1914

Reissued, with supplement, August 29, 1914.

U. S. DEPARTMENT OF AGRICULTURE,
BUREAU OF PLANT INDUSTRY—Circular No. 72.
B. T. GALLOWAY, *Chief of Bureau.*

A MOISTURE TESTER FOR GRAIN AND OTHER SUBSTANCES AND HOW TO USE IT.

BY

J. W. T. DUVEL,

Assistant in Charge of Laboratory Methods,
Office of Grain Standardization.

56644°—14

WASHINGTON : GOVERNMENT PRINTING OFFICE : 1914

BUREAU OF PLANT INDUSTRY.

Chief of Bureau, BEVERLY T. GALLOWAY.
Assistant Chief of Bureau, G. HAROLD POWELL.
Editor, J. E. ROCKWELL.
Chief Clerk, JAMES E. JONES.

B. P. I.—615.

A MOISTURE TESTER FOR GRAIN AND OTHER SUBSTANCES AND HOW TO USE IT.[a]

INTRODUCTION.

The apparatus described in this circular, which is known commercially as the Brown-Duvel moisture tester, was originally described in 1907 in Bulletin 99 of the Bureau of Plant Industry, entitled "A Quick Method for the Determination of Moisture in Grain," and was developed primarily to meet the demands of grain dealers for a rapid and exact method of determining the moisture content of commercial corn. Since that time some slight modifications have been made in both the apparatus and the method, and specific instructions have been worked out for making moisture tests of the more important cereal grains and some of the more important seeds. It has been demonstrated that the method is entirely practicable for making moisture determinations of practically all substances which admit of a free circulation of the oil during the heating.

Both the construction and the process have been made as simple as possible, so that reliable tests can be made by any careful worker, even though he has not had the benefit of special training in a chemical laboratory. It is, however, of the utmost importance that the operator be a careful and honest worker who can be depended upon to follow simple instructions and to report results correctly. It must be borne in mind that the instructions given in this circular are applicable only when used in connection with the moisture tester herein described and illustrated and do not apply to modified forms of testers.

[a] This circular has been prepared in order to provide specific instructions for making moisture determinations of different substances and to bring about more uniformity in the use of the apparatus. The use of this moisture tester with corn has resulted in more attention being paid to the proportion of dry matter contained therein, and already considerable quantities of corn are being sold on a percentage statement of moisture or on grades in which the percentage of moisture is limited, and it is believed that the time is not far distant when this will be the universal practice. The apparatus described was developed by Mr. Edgar Brown and Dr. J. W. T. Duvel, of this Bureau, and has been patented by them in order to secure its free use by the people of the United States.—Wm. A. Taylor, *Acting Chief of Bureau.*

61825°—Cir. 72—10

DESCRIPTION OF THE APPARATUS.

The apparatus consists of a heating chamber divided into compartments so that a number of samples can be tested at the same time, a tank for cold water, through which the condenser tubes pass, and a suitable stand for supporting the heating chamber and cold-water tank, together with numerous accessories, such as thermometers, distillation flasks, graduated measuring cylinders, etc., all of which are illustrated and described somewhat in detail in the following pages, so that no one should have difficulty in setting up the apparatus and in making the tests properly.

Figure 1 shows an external view of a standard six-compartment moisture tester set up ready for use. Each compartment of the heating chamber is numbered and provided with a small mica window, through which the operator can observe the action within the flask while the test is in progress. Figure 2 is a cross section through one of the compartments of the tester, showing the position of the flask and the flask support within the compartment, the adjustment of the thermometer, and the proper connections of the various parts.

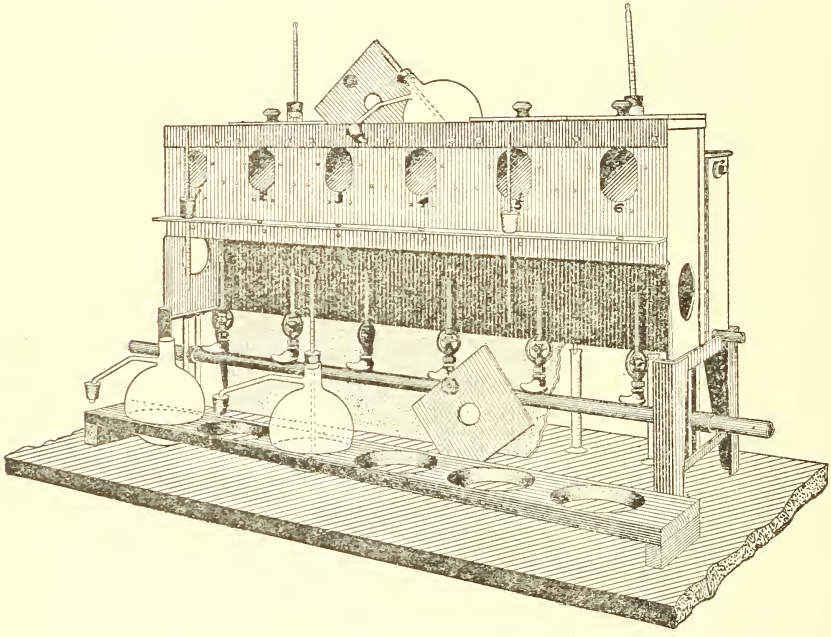

FIG. 1.—Six-compartment tester, with wooden rack in foreground.

Figures 1 and 2 show testers equipped with burners for using gas as fuel. The form of construction, however, is such that alcohol or gasoline burners can be used with equally good results, provided the burners give a pointed rather than a spreading flame and the apparatus is modified in no other way.

THERMOMETERS.

It is of the utmost importance that the thermometers be of extra quality and not the ordinary grade of chemical thermometers which are so commonly furnished with apparatus of this general character. The success of this method of making moisture determinations depends largely upon the accuracy with which the temperature readings are made, and any thermometer showing an error of more than one-half of a degree should not be used unless such errors are known and provided for in the readings.

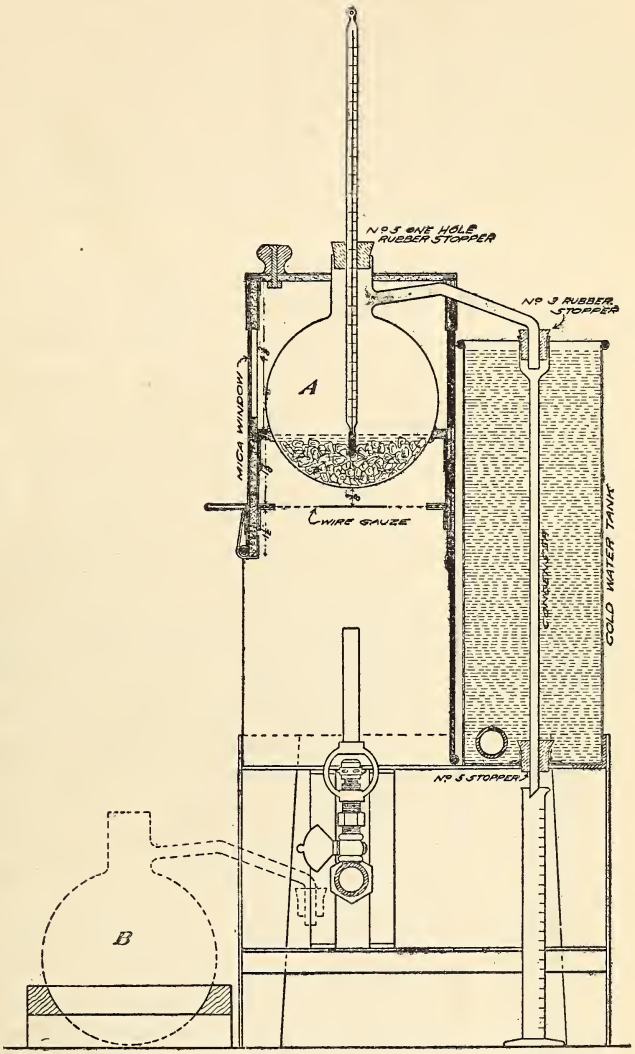

FIG. 2.—Sectional view, showing the various parts properly connected for use. *A*, Distillation flask in position, three-eighths of an inch above the wire gauze; *B*, distillation flask in wooden rack, used only during filling.

It is also necessary that the mercury bulbs of the thermometers be of approximately the same length, so that some uniformity can be had as to the depth to which the thermometers are immersed in the oil, the correct positions of which are shown in figure 9, on page 10.

SPECIFICATIONS FOR THERMOMETERS.

In order to secure a high-class thermometer at a minimum cost it has been found advisable to adopt a thermometer which can be used for general laboratory work. The thermometer should be graduated in whole degrees from 0° to 200° C., with the graduations etched on a stem having a white background. The diameter of the stem of the thermometer should be nine thirty-seconds of an inch, so that it will fit properly in the No. 5, one-hole, rubber stopper. The length of the mercury bulb should be approximately three-fourths of an inch—not greater than thirteen-sixteenths nor less than eleven-sixteenths of an inch. The total length of the thermometer should not exceed 13 inches.

The manufacturers should be required to furnish certificates of correction with all thermometers showing errors in calibration and graduation exceeding one-half of a degree at 175° and 190° C.

GRADUATED MEASURING CYLINDERS.

The small graduated cylinders for measuring the moisture should have a capacity of at least 25 cubic centimeters. They should be carefully graduated in fifths, so that the readings can readily be made in tenths. When 100 grams are used for the test, the number of cubic centimeters of water in the graduate corresponds to the percentage of moisture originally contained in the sample. If only 50 grams are used, as is necessary with some substances, the number of cubic centimeters of water in the graduate is equivalent to one-half of the moisture percentage.

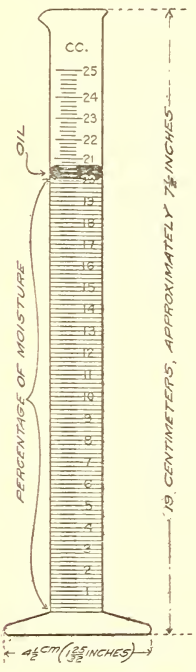

FIG. 3.—Graduated measuring cylinder, showing 20 per cent of moisture.

In making moisture determinations a small quantity of oil—usually less than one-half of a cubic centimeter—is carried over into the measuring cylinder and collects on the surface of the water, so that the readings should be made at the bottom of the meniscus between the oil and the water, as shown in figure 3. Should the oil and the water not separate readily, the graduated cylinder should be whirled by rolling quickly between the two hands.

FIG. 4.—Swab or test-tube cleaner.

After each test the cylinders must be cleaned and dried, which can best be done with a swab or test-tube cleaner having a small sponge attached to one end, as shown in figure 4.

Satisfactory cleaners can also be made as needed by twisting cotton waste, strips of cheese cloth, or some other similar substance about one end of a piece of fairly heavy wire.

DISTILLATION FLASKS.

The special distillation flasks, with a capacity of approximately 1,000 cubic centimeters, should be made of the best grade of resistant glass and should be well annealed in order to withstand sudden changes in temperature without breaking. The necks of the flasks should be sufficiently heavy to stand tight corking. Figure 5 shows one of these flasks in which the dimensions of the different parts are given in both centimeters and inches.

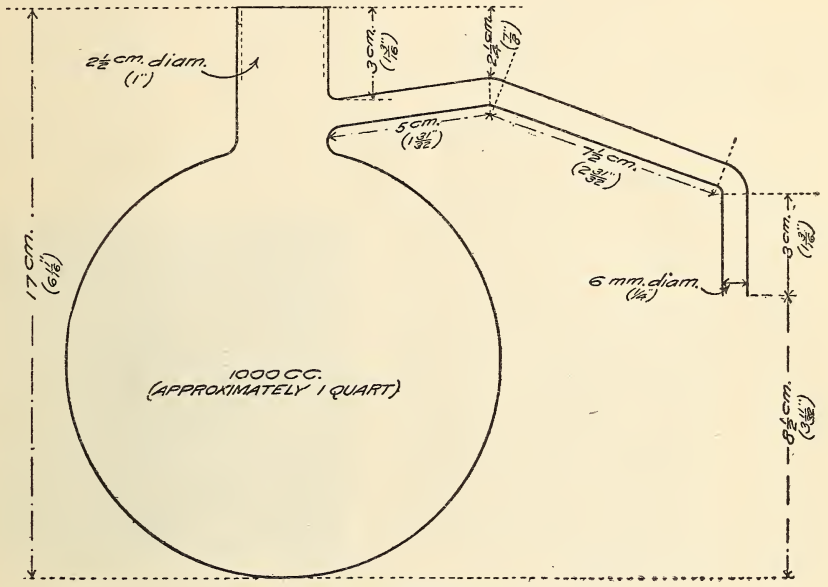

FIG. 5.—Distillation flask.

In some instances dealers have supplied flasks showing such wide variations in the dimensions that they were entirely useless. It is therefore recommended that in ordering flasks it be definitely stated that they must be in accordance with the specifications given in this circular. In case the dimensions of the flasks, as well as other parts of the apparatus, are not approximately as herein specified, they should be returned.

FLASK SUPPORTS.

The pipestem triangles originally used for supporting the flasks above the wire gauze resulted in the breakage of an undue number of flasks, and for that reason these triangles have been replaced with

removable supports, as shown in figures 2 and 6. This form of support provides a good asbestos ring on which the flask rests, exposes the bottom of the flask more uniformly to the action of the heat, and at the same time is so made that a sufficient quantity of heat comes in contact with the upper part of the flask.

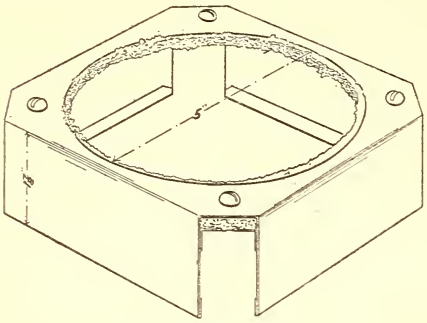

FIG. 6.—Flask support.

In adjusting these supports the asbestos ring in the top of the support should be cut so that the bottom of the flask will be not less than three-eighths of an inch above the asbestos center of the wire gauze, as shown in figure 2. The support should be $1\frac{7}{8}$ inches high.

WIRE GAUZE.

The wire gauze in the bottom of each compartment between the flask and the flame should be kept in good condition, so that the flame does not play directly on the bottom of the flask. This gauze should be made of iron wire, from sixteen to twenty thousandths of an inch in diameter, with 20 wires to the inch and with a 2-inch asbestos center, as shown in figure 7.

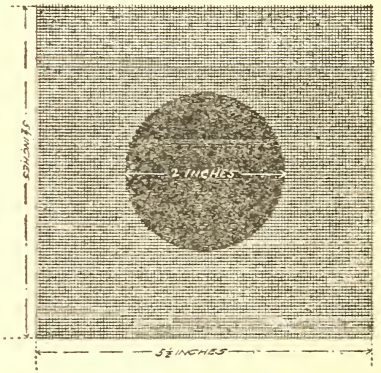

FIG. 7.—Wire gauze with asbestos center.

The asbestos center insures a more uniform distribution of heat and adds greatly to the life of the wire. It can readily be put in by mixing asbestos cement with water and rubbing it into the mesh of the gauze.

CONDENSER TUBES.

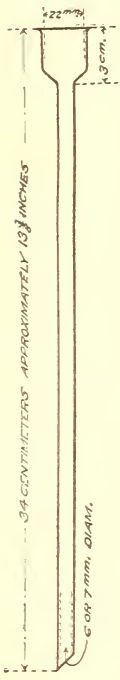

FIG. 8.—Condenser tube.

The glass condenser tubes, with dimensions as shown in figure 8, should be so adjusted in the No. 5 rubber stoppers that when the latter are firmly pressed into the holes in the bottom of the cold-water tank the tops of the tubes will be approximately one-fourth of an inch above the top of the tank.

RUBBER STOPPERS.

The No. 5, one-hole, rubber stoppers which carry the thermometers and the No. 3, one-hole, rubber stoppers used on the side tubes of the flasks should be of such quality as will withstand comparatively high temperatures and resist the action of the oil. Stoppers containing a large percentage of pure gum are not desirable. They soon swell and become unfit for use. Before attempting to put any of the glass parts through the holes in the rubber stoppers they should be moistened with oil or water, thereby lessening the chances of breaking the tubes and cutting the hands.

GENERAL DESCRIPTION OF THE METHOD.

The method consists in heating whole grains, seeds, spices, or other similar substances in a mineral oil having a flashing point much above the boiling point of water, in condensing the water which distills off, and in collecting and measuring it in a suitable graduate. The method, as already stated, is so simple that the tests can be made by any careful worker who is able to follow simple instructions.

A single moisture determination can be made in twenty-five or thirty minutes, and with a 6-compartment tester, as shown in figure 1, six tests can be made in approximately the same time. In commercial work, where a large number of tests are to be made, one man and a helper, with the use of three 6-compartment machines, can readily make 200 or more tests in a day of eight hours, an average of less than two and one-half minutes for each test. In addition to the rapidity with which moisture determinations can be made by this method, it is especially valuable in testing substances which have a large percentage of water, are difficult to grind, or contain such large quantities of volatile oils that determinations based upon differences in weight during drying are unreliable. This method also obviates the grinding of samples, thus eliminating the loss of water which accompanies the grinding of material of relatively high moisture content. Moreover, ground material has a tendency to "cake" in the flask; consequently, this method is not entirely satisfactory for making moisture determinations of flour, meal, powders, or other similar substances, without some further modification.

HOW TO MAKE A TEST.

After properly mixing the bulk sample, carefully weigh the desired quantity for the moisture test and empty immediately into the distillation flask, to which add the oil and shake, with a slight whirling motion, until the two become well mixed. Grasp the neck of the flask in one hand and hold it in such a manner that when the No. 5

rubber stopper carrying the thermometer is inserted it can be readily determined whether the mercury bulb of the thermometer is properly adjusted in the oil, as shown in figure 9. Place the flask in the proper compartment of the tester and proceed in a similar manner with the remaining samples.

The correct adjustment of the thermometer is a factor which must not be ignored. If the mercury bulb of the thermometer is too near the bottom of the flask the percentage of moisture will be too low. For the same reason it is important that there be some uniformity in the length of the mercury bulbs of the thermometers, which should be approximately three-fourths of an inch.

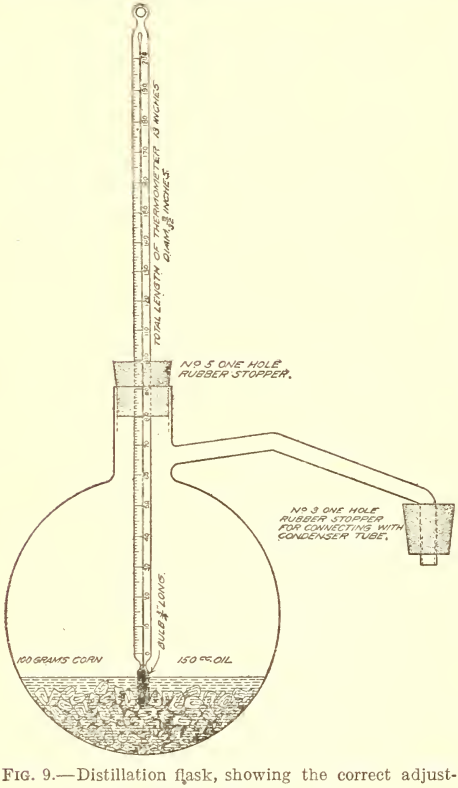
TOTAL LENGTH OF THERMOMETER 12 INCHES
DIAMETER 5/8 INCH
No. 5 ONE HOLE RUBBER STOPPER.
No. 3 ONE HOLE RUBBER STOPPER FOR CONNECTING WITH CONDENSER TUBE.
BULB 3/4 INCH
100 GRAMS CORN
150 cc. OIL

FIG. 9.—Distillation flask, showing the correct adjustment of the thermometer.

As the thermometer is being inserted, glance along the side tube of the flask to make certain that it has not become stopped by the lodgment of some particle while emptying or filling the flask. If the side tube is not open to permit the free escape of the rapidly forming steam the pressure during the heating will become sufficient to blow out the stopper and thermometer, or possibly to burst the flask.

When the flasks have been filled and placed in the compartments of the tester, connect the side tubes of the flasks by means of the No. 3 rubber stoppers with the thimbles of the glass condenser tubes which extend down through the cold-water tank, so that the moisture which is liberated from the grain or other substance will be condensed and collected in the graduated cylinders beneath the tank. Figure 2 shows the correct manner of adjusting the various parts of the apparatus.

Place the cover over the flask, then ignite the gas, adjusting the flame so that it will require about twenty minutes to reach the temperature prescribed for the substance being tested. When the desired

temperature is reached extinguish the flame quickly, after which the thermometer will show a slight gradual increase in temperature. If the sample contains a large percentage of moisture there is danger of boiling over, but this can usually be prevented by lowering the flame whenever the action in the flask becomes too vigorous, as can be readily determined through the mica window in the front of the compartment. As soon as the water stops dropping from the condenser tube, which usually requires from four to six minutes after the flame has been extinguished, the test is complete. However, before reading the amount of moisture remove the covers and then disconnect the flasks from the condenser tubes in order to allow the small quantity of moisture which sometimes collects at the base of the No. 3 rubber stoppers to drop into the graduated measuring cylinders. The percentage of moisture is read beneath the layer of oil on top of the water, as shown in figure 3. In order to guard against possible error it is desirable to make duplicate tests of all samples and take the average of the two readings as the correct percentage of moisture.

While the contents of the flasks are still hot, take the flask by the side tube and after giving a slight whirling motion invert quickly, emptying the contents into a suitable strainer, so that the oil can be recovered for further use.

When the flasks are not in use, keep them in place in the compartments and make all connections the same as for a test. In using a new flask for the first time "run" a preliminary sample previous to making a regular test, so that all the flasks will be in uniform condition.

HOW TO TEST DIFFERENT SUBSTANCES.[a]

Detailed instructions have been worked out for making moisture determinations of a limited number of substances, as follows:

Barley.—Use 100 grams of grain and 150 cubic centimeters of oil, and extinguish the flame when the thermometer registers 190° C.

Corn (maize).—Use 100 grams of grain and 150 cubic centimeters of oil, and extinguish the flame when the thermometer registers 190° C. This method, however, can not be used with the pop corns.

[a] Different substances are likely to require slightly different treatment, which can be determined only by careful experiments covering a wide range of conditions, checking the results with the tests made by drying a definite quantity to constant weight and calculating the loss in weight as the percentage of moisture. The data here shown were secured by checking the results with moisture determinations made by drying to constant weight in the common type of double-walled oven filled with water maintained at the boiling point, except that the proper treatment for flaxseed was determined by checking with samples dried to constant weight in a 28-inch vacuum at a temperature of 99° to 100° C. Moisture determinations made in vacuum at temperatures of 105° or 108° C. will show higher percentages than will result from the method herein described.

Corncobs.—Use 50 grams of cob cut in pieces that can be easily removed from the flask and 250 cubic centimeters of oil, and extinguish the flame when the thermometer registers 190° C.

Cottonseed.—Use 50 grams of seed and 150 cubic centimeters of oil, and extinguish the flame when the thermometer registers 190° C.

Flaxseed.—Use 100 grams of seed and 150 cubic centimeters of oil, and extinguish the flame when the thermometer registers 175° C.

Kafir.—Use 100 grams of grain and 150 cubic centimeters of oil, and extinguish the flame when the thermometer registers 190° C.

Oats.—Use 50 grams of grain and 150 cubic centimeters of oil, and extinguish the flame when the thermometer registers 195° C. In oats that are light and chaffy special care should be taken to insure the thorough mixing of the oil and grain and to heat with a slower flame in order to reduce the foaming to a minimum. In extreme cases it may be necessary to add an extra 50 cubic centimeters of oil.

Rice (unhulled).—Use 100 grams of grain and 150 cubic centimeters of oil, and extinguish the flame when the thermometer registers 190° C.

Rye.—Use 100 grams of grain and 150 cubic centimeters of oil, and extinguish the flame when the thermometer registers 175° C.

Soy bean.—Use 100 grams of beans and 150 cubic centimeters of oil, and extinguish the flame when the thermometer registers 170° C.

Wheat.—Use 100 grams of grain and 150 cubic centimeters of oil, and extinguish the flame when the thermometer registers 180° C.

OIL FOR THE MOISTURE TESTS.

In making tests a good grade of mineral engine oil should be used. The oil must be free from water, should have a flashing point in open cup of approximately 200° to 205° C. (392° to 401° F.), and preferably a viscosity between 10 and 15 (Engler) at 20° C. (68° F.). The higher the viscosity the greater the danger of boiling over, and for this reason cylinder oils should not be used; as a rule, they are too heavy for good results.

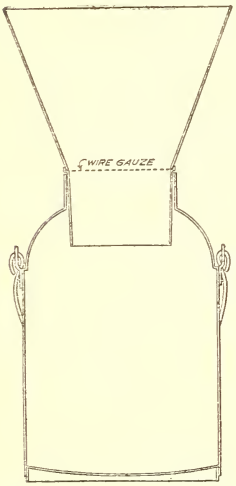

FIG. 10.—Strainer for recovering the oil.

After the tests are completed and while the oil is still hot, empty the contents of the flasks into a strainer to recover the oil, which can be used repeatedly. A funnel strainer fitted to the mouth of a 3 or 4 gallon milk can, as shown in figure 10, is serviceable and inexpensive.

MEASURING THE OIL.

A conical glass graduate, as shown in figure 11, having a capacity of 150 cubic centimeters, is ordinarily used for measuring the oil for the test. A more complete device consists of a

graduated glass gauge and a stopcock fitted to a cylindrical brass cup of a size to hold sufficient oil for at least a dozen tests, as shown in figure 12.

DRAWING AND HANDLING SAMPLES.

It is admitted that to secure a representative sample is often very difficult, but the necessity of sparing no trouble in this respect is most strongly urged.

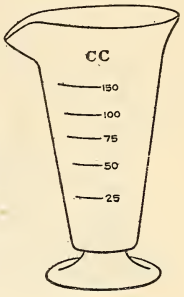

FIG. 11.—Oil measure.

In securing samples of grain from cars, etc., it must be remembered that, as a rule, grain from the surface will show less moisture than that in the body of the shipment. In corn in cars it is not uncommon to find differences of several per cent between the moisture content of the layer of corn immediately at the surface and that of the body of the grain directly beneath. In an experimental shipment of three lots of corn contained in a single car in which special care was taken to see that each lot was uniform throughout, moisture tests at destination, fifteen days after shipment, showed 14.1 per cent, 14.3 per cent, and 14.1 per cent for corn from the surface of each of the three lots, as compared with 17.3 per cent, 16.9 per cent, and 19.2 per cent, respectively, for the samples taken with a grain trier from 2 to 3½ feet below the surface, the latter percentages being practically the same as at the time of shipment.

FIG. 12.—Improved oil measure. Each number on the glass gauge represents 150 cubic centimeters of oil.

Likewise, the same care must be observed in handling the samples after they are drawn from the bulk or weighed for the test. If exposed to the air they will lose moisture in accordance with the condition of the atmosphere and the percentage of water in the sample. A preliminary test with 100 grams of wheat containing 14.5 per cent of moisture showed a loss of 0.20, 0.35, 1.10, and 1.90 per cent when exposed to the air of the laboratory for periods of one-half, one, four, and twenty-six hours, respectively. A second lot of wheat containing 16.1 per cent of moisture lost 0.50, 0.75, 1.75, and 3.20 per cent, respectively, during the same periods under identical treatment.

BALANCE FOR WEIGHING SAMPLES.

While not a part of the apparatus, a balance is necessary for weighing the samples. One of simple construction, having a sensitiveness of one-twentieth or even one-tenth of a gram has been found quite

satisfactory. Balances similar to the two types shown in figure 13, which have a special scoop for emptying the weighed samples into the distillation flasks, will meet all requirements. Such balances are likewise much less expensive, less likely to get out of order, and more easily operated than the delicate analytical balances commonly used in chemical laboratories.

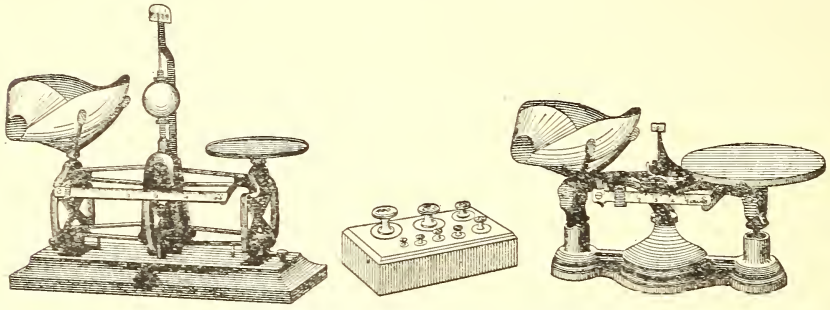

FIG. 13.—Balances with a set of polished brass weights, 400 grams to 5 grams. Left, torsion balance; right, trip scale with agate bearings.

Whatever the type of balance used, it should rest on a firm support, preferably a heavy shelf securely fastened to a solid wall of the building, which will greatly facilitate the keeping of the scale in balance, which is absolutely essential for reliable work.

SPECIAL POINTS FOR CONSIDERATION.

(1) The moisture tester should be installed in a place where it will not be exposed to strong air currents.

(2) The tester can be equipped for burning alcohol or gasoline, but gas is to be preferred whenever available.

(3) The wire gauze with asbestos center should be kept in good condition so that the flame will not play directly upon the bottom of the flask.

(4) The bottom of the flask should be not less than three-eighths of an inch above the wire gauze.

(5) See that the column of mercury in the thermometer is continuous; if broken it should be shaken down.

(6) Adjust the thermometers in the rubber stoppers so that the bulbs of the thermometers will be approximately four-fifths immersed in the oil.

(7) Keep a good supply of cold water running through the condenser tank.

(8) Adjust the flame so that about twenty minutes are required to reach the prescribed temperature.

(9) If the moisture content of the sample is high, so that there is a tendency to boil over, lower the flame until a considerable portion of the water is distilled over.

(10) When the thermometer registers the prescribed temperature, extinguish the flame promptly.

(11) After the flame is extinguished a slight gradual increase in temperature is to be expected. A sudden increase or a sudden decrease in temperature of several degrees indicates that the flame was too intense during the latter part of the heating and the test should be repeated.

(12) See that there is no water in the graduated cylinder before starting the test.

(13) Read the percentage of moisture in the graduated cylinder beneath the layer of oil on top of the water.

(14) If the water which distils over is discolored, the substance has evidently been burned and the test should be repeated.

(15) When not in use keep the flasks in position in the compartments, with all connections made as for making a test.

(16) Before making a test in a new flask, "run" a preliminary sample, so that all the flasks will be in uniform condition.

(17) Draw all samples carefully and keep them in air-tight containers until ready to make the test. They lose moisture rapidly when exposed to the air.

(18) Place the scales on a firm support and see that they are in balance before making a weighing.

(19) The specific instructions for making tests given in this circular do not apply to modified forms of testers.

Approved:

JAMES WILSON,
Secretary of Agriculture.

WASHINGTON, D. C., *September 16, 1910.*

[Cir. 72.]

SUPPLEMENT.

INSTRUCTIONS FOR TESTING BARLEY MALT AND DISTILLERS' DRIED GRAINS.

Detailed instructions for making moisture determinations of barley, corn (maize), corn cobs, cotton seed, flaxseed, kafir, oats, rice (unhulled), rye, soy beans, and wheat are given on pages 11 and 12 of this circular. Since this circular was first published the investigations have been continued and the following instructions for making moisture determinations of barley malt and distillers' dried grains should be added to the foregoing list.

Barley Malt.—Use 100 grams of malted barley and 200 cubic centimeters of oil, and extinguish the flame promptly when the thermometer registers a temperature of 168° C. The flame should be adjusted so that it will require approximately 20 minutes to reach the prescribed temperature.

Distillers' Dried Grains.—Use 50 grams of distillers' dried grains and 200 cubic centimeters of oil, and extinguish the flame promptly when the thermometer registers a temperature of 190° C. Special care should be taken to see that the oil and the distillers' dried grains are thoroughly mixed before beginning the test.

THE USE OF COPPER VS. GLASS FLASKS.

Numerous requests have been received from time to time as to the advisability of using copper instead of glass flasks for making moisture determinations by the method described in this circular. The results of investigations by J. H. Cox, Assistant in Grain Standardization, have shown that copper flasks may be used in the tester illustrated in figures 1 and 2, although glass flasks have proved more satisfactory for careful work. In making moisture determinations of corn and oats, the same treatment is required in the use of copper flasks, when spun from 16-ounce sheet copper and used in the tester described in this circular, as is required when the tests are made in glass flasks, the essential feature being to extinguish the flame promptly when the thermometer reaches a temperature of 190° C. for corn and 195° C. for oats. However, in using copper flasks in this machine for making moisture determinations of wheat the flame should be extinguished when the thermometer reaches a temperature of 185° C. and not at 180° C., as is required when using glass flasks, otherwise the treatment should be the same. Correct temperatures for use with copper flasks in making moisture determinations of the other grains have not been determined.

SPECIAL FLASK FOR TESTING FLOUR AND MEAL.

Mr. Cox has likewise designed a special double-walled flask for use in connection with this tester for making moisture determinations of flour and meal. This flask and details for determining the moisture content of flour and meal are fully described in U. S. Department of Agriculture Bulletin No. 56, issued January 28, 1914.

July 25, 1914.

○